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Applicants : Masaaki OYAMADA et al. Art Unit: 1792
Application No.: 10/820,024 Examiner: Tsoy, E.
Filing Date: April 8, 2004
Title : CONDUCTIVE ELECTROLESSLY PLATED POWDER AND METHOD FOR MAKING SAME

Commissioner for Patents
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DECLARATION UNDER 37 CFR 1.132

Sir:

I, Shinji Abe, do declare and state as follows:

I am one of the applicants of the above-identified application;

I have been employed by NIPPON CHEMICAL INDUSTRIAL CO., LTD for 10 years as a researcher in the field of conductive electroless plated powders;

I have read all of the Office Actions in the above-entitled application, and have read and am familiar with each of the references cited in the Office Actions by the Examiner; and

The following experiments were carried out by me or under my direct supervision and control and the results are true and correct to the best of my knowledge.

I. Objects

Experiment 1

Experiment 1 relates to using claimed step (III) the aqueous suspension containing the core powder obtained from Experiment 2 of the Declaration filed on October 26, 2007. (Experiment 2 of the Declaration filed on October 26, 2007 previously repeated the steps described in Kawakami et al. (JP 1-242782)).

The purpose of this experiment is the following:

To verify that no columnar structures extending in a direction of the thickness of the nickel film were formed on the surface of a core powder using claimed step (III) on the aqueous suspension containing the core powder obtained from Experiment 2 of the Declaration filed on October 26, 2007.

Experiment 2

Experiment 2 relates to conductive electroless plated powders used in the present invention and Weber et al. (U.S. Patent No. 6,274,241).

The purpose of this experiment is the following:

To verify that no nickel film was formed on the surface of a core powder using claimed step (III) from the

present invention on the aqueous suspension prepared by the method of Weber et al.

II. Brief Description of the Drawings

FIG. 1 is an SEM photograph using a magnification of 100,000 showing a plating powder obtained in Experiment 1.

FIG. 2 is an optical microscope photograph (magnification of transmitted light: 100) showing a plating powder obtained in Experiment 2.

III. Experiments

(1) Experiment 1

The steps described in Experiment 2 of the Declaration filed on October 26, 2007 are repeated below

According to the description at page 7, lower right column, line 17 to page 8, upper left column, line 19 of Japanese Unexamined Patent Application Publication No. 1-242782 (see page 22, line 19 - page 23, line 22 of the translation), catalyzing treatment was performed using palladium on the surface of a spherical phenol resin serving as a core powder. The wastewater of electroless plating after use in Experiment 1 (1,000 ml) of the Declaration filed on October 26, 2007, was added to an aqueous suspension containing the core powder subjected to the catalyzing treatment, and formation of an initial thin film of nickel was attempted (as discussed in the result of Experiment 2 in the Declaration filed on October 26, 2007, the initial thin film of nickel was not formed on the surface of the core particles).

Electroless Plating Step

According to the description set forth in line 9 of page 21 to line 6 of page 22 in the present invention (similar to claimed step (III)), two solutions, i.e., the nickel ion-containing solution (b) and the reducing agent-

containing solution (c) shown in example 1 of Table 1, were individually and simultaneously added to the aqueous suspension prepared in the above described step. The adding rate of the nickel ion-containing solution (b) and the reducing agent-containing solution (c) is shown in Table 1 of the present invention. The volume of each solution added was 870 ml. Immediately after the addition of the two solutions, generation of hydrogen was observed, and the start of a plating reaction was confirmed. Until the addition of the two solutions was completed, the concentration of the amino group-containing complexing agent in the aqueous suspension was maintained at the value shown in example 1 of Table 1. After the completion of the addition of the two solutions, stirring was continued while maintaining the temperature at 75 °C until bubbling of hydrogen was stopped. The load after the completion of the addition of the two solutions was 2.4 m^2/l. The aqueous suspension was then filtered, and the filtrate was subjected to repulping-washing three times, followed by drying with a vacuum dryer at 110 °C.

(2) Experiment 2

Preparation of Catalyzation Particles

Catalyzation particles were prepared in accordance

with Example 1 of Weber et al.

Glass powder with particle sizes in the range between approximately 60µm and 200µm was digested with dilute hydrofluoric acid to roughen the surface, washed until neutral and dried. Five grams of the powder were placed in 0.5 L of a 10% solution of allyldimethylchlorosilane in chloroform at room temperature. The mixture was stirred overnight at room temperature, whereupon the reaction illustrated by the first reaction equation above took place. Then the solution was separated, and the powder was washed with chloroform and then dried. The dried powder was treated with 0.5 L of a 10% solution of the cyclooctadiene complex of palladium chloride in chloroform for 12 hours at room temperature while stirring.

Next the solution was separated, and the powder was washed with chloroform and then dried. Examination under the microscope showed that the powder surface was uniformly colored, indicating that the nucleation was uniform, i.e., uniformly thick and mostly closed.

Preparation of the Plating Bath

The plating bath was prepared as described in column 7, lines 26-40 of Weber et al.

The plating bath solution was composed of

49.5g Na_2WO_4,

65.4g Na gluconate,

6.57g $NiSO_4 \cdot 6H_2O$,

5.13g dimethylaminobrane,

50mg thiourea,

approx. 500 ml H_2O, and

approx. 75 ml of a 25% ammonia solution.

The plating bath had a pH of 9, was heated to 80°C and stirred.

Electroless Plating Step

10 g of core particles that were obtained as described in the catalyzation particles producing step were added in 100 ml H_2O and formed a slurry solution. Thereafter, its temperature was increased to 80°C. As soon as, 500 ml of the above plating bath solution was adjusted, it was added in the slurry solution quickly and an aqueous suspension was provided. After the completion of the addition of the plating bath solution, stirring was continued while maintaining the temperature at 80°C for 10 minutes.

Two solutions, i.e., the nickel ion-containing solution (a) and the reducing agent-containing solution (b) shown in Table 1 of example 1 of the present invention, were individually and simultaneously added to the aqueous suspension prepared in the above-operation each at the

adding rate shown in the Table 1 of example 1 of the present invention. The volume of each solution added was 800 ml.

Then, the plating bath was filtered and the particles were subjected to repulping-washing with water and the particles were vacuum-dried at 110 °C. The particles obtained are shown in Fig. 2.

IV. Result and Discussion

The result of Experiment 1:

The electroless nickel plated powder obtained in Experiment 1 was embedded in an epoxy resin, followed by curing. Then, the resin was cut to expose a cross section. Using this section as a measuring object, the cross section in the thickness direction of the nickel film of the electroless nickel plated powder was observed with a SEM. The result thereof is shown in Fig. 1 below. The SEM image shown in Fig. 1 has a magnification of × 100,000. As is evident from the result shown in Fig. 1, the nickel film was composed of an aggregate of many crystal grains. However, the columnar structures extending in a direction of the thickness of the nickel film were not recognized.

The result of Experiment 2:

After adding the metal solution to the slurry solution, the pH was changed and this caused the metal solution to decompose.

After that, a nickel ion-containing solution (a) and the reducing agent-containing solution (b) were individually and simultaneously added to the above aqueous suspension, no metal reaction occurred on the core particles because an electroless plating reaction is generally carried out on an activity place. On the contrary, there was no initial thin film and therefore the electroless plating reaction was not carried out.

Optical microscope photographs of the plating powders produced in Experiment 2 shown in Fig. 2.

Since nickel metal was not disposed on the surface of the core particles in Experiment 2, the light from the optical microscope went through the particles. Therefore, since almost all of the plating powders shown in Fig. 2 are not completely coated, they are shown as being transparent in this figure. We also confirmed that decomposed fine nickel metal powders existed around the core particles.

V. Conclusion

As discussed above and shown in Fig. 1, in the plating powders produced by Experiment 1, the columnar structures extending in a direction of the thickness of the nickel film were not recognized.

As discussed above and shown in Fig. 2, the nickel metal was not coated on the surface of the core particles produced by Experiment 2.

I further declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: April 30, 2008

Shinji Abe

Shinji ABE

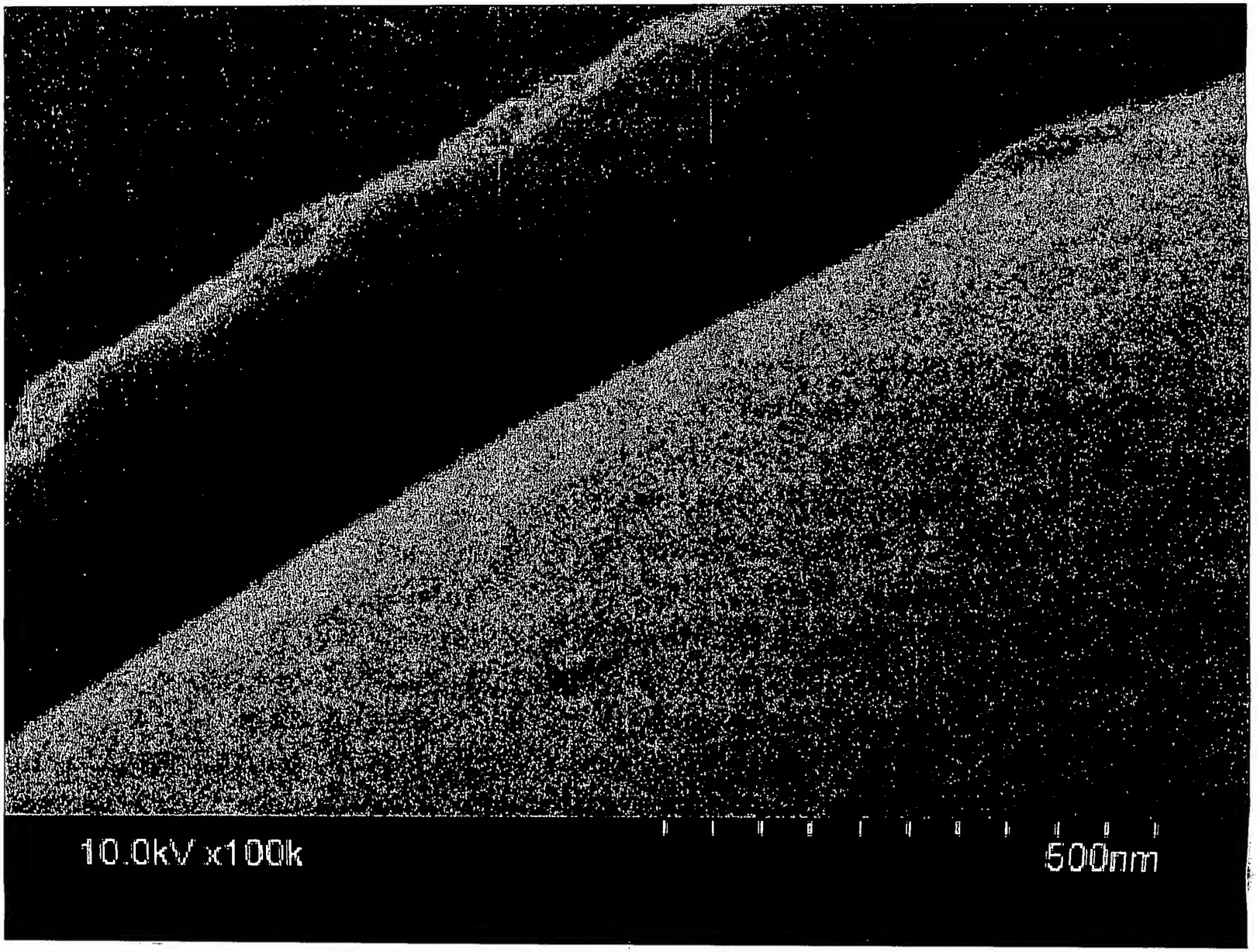

Fig. 1

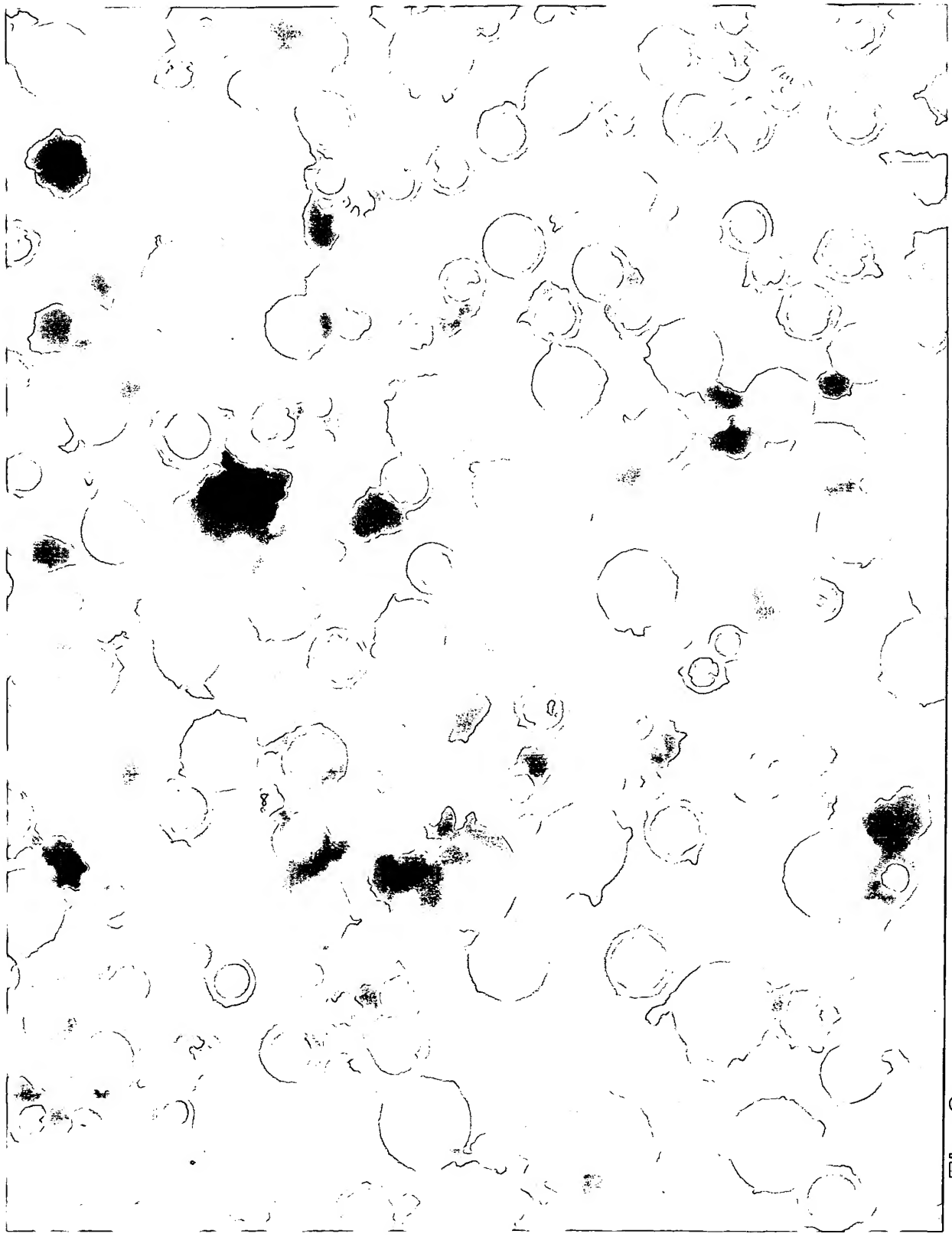

Fig. 2